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<p align="center">Division of Forensic Science</p> <p align="center">TRACE EVIDENCE PROCEDURES MANUAL</p>	<p align="center">Amendment Designator:</p>
	<p align="center">Effective Date: 31-March-2003</p>
<p align="center">10 GUNSHOT RESIDUE ANALYSIS</p> <p>10.1 Analytical Approach</p> <p>Generate one or more Gunshot Residue (GSR) Evidence Handling Worksheets (Appendix 19). For FACTS results data set purposes, each GSR kit is to be considered a separate item. Open the GSR kit and label each of the sample vials. Visually examine stub surface. If debris is present go to ¶ 10.2, Carbon Evaporation. Otherwise, proceed to ¶ 10.3, 10.4 or 10.5, Automated Scanning Electron Microscope/Energy Dispersive X-ray System (SEM/EDS) analysis.</p> <p>10.1.1 Minimum Standards and Controls</p> <p>10.1.1.1 GSR kits are ordered by DFS to our specifications. When a lot of GSR kits arrives, two percent of the kits must pass QC inspection before any kits from that lot are released to User Agencies.</p> <p>10.1.1.2 A visual inspection is made of the GSR kits to note whether all components of the kit are present. Any visible debris on the collection surface is noted. One sample from each kit is run by automated SEM/EDS analysis. A copy of the GSR SEM/EDS Worksheet and stats file is retained. Samples from these kits become future negative control samples for automated GSR runs.</p> <p>10.2 Carbon Evaporation</p> <p>10.2.1 Purpose</p> <p>10.2.1.1 Carbon evaporation, or coating, makes the sample electrically conductive and reduces charging in the SEM. In addition, carbon does not interfere with EDS analysis.</p> <p>10.2.1.2 <u>Special Considerations that should be noted with this technique:</u> Carbon evaporation reduces charging on samples containing visible debris. Therefore, the overall automated SEM/EDS run time may be shortened due to reduction in charging of the sample.</p> <p>10.2.2 Safety Considerations</p> <p>10.2.2.1 Carbon evaporation produces a bright arc at the carbon rod tip source. During evaporation, the carbon rod should only be viewed through an appropriate welder's glass.</p> <p>10.2.3 Minimum Standards and Controls</p> <p>10.2.3.1 A negative control is included with all GSR samples to be coated for a given run. If a single sample requires carbon coating then all samples in that particular GSR automated run will be coated.</p> <p>10.2.3.2 A glass slide and coverslip are used to evaluate the coating.</p> <p>10.2.4 Analytical Procedures</p> <p>10.2.4.1 Label the bottom of the sample stubs before coating samples.</p> <p>10.2.4.2 Operate the carbon evaporator following the manufacturer's operations manual.</p> <p>10.2.4.3 Slowly rotate the sample plenary stage during the coating procedure.</p> <p>10.2.5 References</p> <p>10.2.5.1 Gabriel, B. L. SEM: A User's Manual for Materials Science. 1985, pp.156 -161. ISBN: 0-87170-202-9.</p>	

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<p>10.3 Automated SEM/EDS GSR Analysis, Zeiss/Oxford Systems</p> <p>10.3.1 Purpose</p> <p>10.3.1.1 Automated SEM/EDS is used to search samples for potential primer residue particles. This instrument searches 500 fields at a magnification of 300X on each sample. Generally speaking, a primer residue particle is defined as a molten or spherical particle containing the elements: Pb, Ba and Sb. Particles indicative of primer residue would have similar morphology and include 2 of the 3 elements listed above.</p> <p>10.3.1.2 This is a sensitive, non-destructive technique that is specific for gunshot residue.</p> <p>10.3.2 Safety Considerations</p> <p>10.3.2.1 The EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling any dewar.</p> <p>10.3.2.2 Be aware of elevated temperatures when changing a filament which has been in operation.</p> <p>10.3.3 Minimum Standards and Controls</p> <p>10.3.3.1 The EDS detector is calibrated before every GSR run and every manual review. The detector resolution must be $\leq 155\text{eV}$ FWHM on cobalt at 1500 cps - 2000 cps, 20 kV, 25mm WD. If this resolution can't be achieved the detector must be conditioned. After conditioning the detector must be autocalibrated.</p> <p>10.3.3.2 Before an automated GSR run can begin the SEM filament must be saturated at 25 kV and beam drift should be less than 1% as measured by the difference in the net window integral for Co on two consecutive 100 second acquisitions. To ensure an adequate count rate the net integral must be between 75,000 and 125,000 counts for a 100 second acquisition.</p> <p>10.3.3.3 A positive control consists of a sampling device containing known primer residue particles and iron spheroids.</p> <p>10.3.3.4 The backscatter detector threshold is set to detect GSR particles ≥ 1 micron in diameter.</p> <p>10.3.3.5 In an automated SEM/EDS GSR run the same area on the positive control is analyzed at the beginning of the run, after 3 samples have been examined and at the end of the run. The number of GSR particles detected during the second of two consecutive positive control fields must not fall below half of the original number of particles detected on the first positive control field.</p> <p>10.3.3.6 In every automated SEM/EDS GSR run, a negative control is analyzed just before the final positive control field. Five hundred fields at 300X are analyzed on the negative control sample. The negative control is the first sample to be loaded in the SEM and the last sample to be removed after the run is complete. Negative control samples can be reused providing they have not been carbon coated. On or about the 1st of each month the current negative control sample will be discarded. A carbon coated negative control sample will be discarded after the automated GSR run due to loss of adhesive properties during the carbon coating procedure.</p> <p>10.3.3.7 Approximately once a month the photographic unit of the SEM is checked using an ASTM measurement standard and a gold standard on carbon. Apertures are replaced if the image can't be stigmated.</p> <p>10.3.4 Analytical Procedures</p> <p>10.3.4.1 The SEM is pumped down to a vacuum of 10^{-5} hPas.</p>	

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<p>10.3.4.2 The filament is saturated at 20 kV and a gain calibration is performed on the EDS detector.</p> <p>10.3.4.3 The SEM is set up for automated GSR run as described in the "GSR Worksheet SEM/EDS" (Appendix 19).</p> <p>10.3.4.4 The instrument is allowed to stabilize for at least 15 minutes.</p> <p>10.3.4.5 Beam drift check is performed as described in Minimum Standards and Controls (§ 10.3.3).</p> <p>10.3.4.6 All files with extensions: .BM, .VD, .VR, .VT are erased from the hard drive. All .SU files are erased <u>except</u> \$____.SU. All .FC files are erased from the hard drive except FSGSR.FC, DEMO.FC.</p> <p>10.3.4.7 The prerun sequence is run.</p> <p>10.3.4.8 During prerun sequence the backscatter electron detector threshold is set to detect particles ≥ 1.0 micron in diameter.</p> <p>10.3.4.9 The prerun sequence prints out the number of GSR particles detected in the positive control field. The operator verifies that 1.0 micron diameter GSR particles are identified.</p> <p>10.3.4.10 The NC6NOBS sequence is run.</p> <p>10.3.4.11 The backscatter detector threshold is set as described earlier.</p> <p>10.3.4.12 The filenames for the samples to be analyzed are input in the order shown on the "GSR Worksheet SEM/EDS".</p> <p>10.3.4.13 The automated run is started.</p> <p>10.3.4.14 When the automated run is finished (typically the following day) a gain calibration is run on the EDS detector as described earlier.</p> <p>10.3.4.15 The SEM/EDS parameters are set up as listed on the "GSR Worksheet SEM/EDS".</p> <p>10.3.4.16 The operator performs a manual confirmation of the potential GSR particles detected during the automated run.</p>	
<p>10.4 Automated SEM/EDS GSR Analysis, R. J. Lee Instrument (PSEM)</p> <p>10.4.1 Purpose</p> <p>10.4.1.1 Automated SEM/EDS is used to search samples for potential primer residue particles. This instrument searches a user defined area at a magnification of 500X on each sample. Generally speaking, a primer residue particle is defined as a molten or spherical particle containing the elements: Pb, Ba and Sb. Particles indicative of primer residue would have similar morphology and include 2 of the 3 elements listed above.</p> <p>10.4.1.2 This is a sensitive, non-destructive technique that is specific for gunshot residue.</p> <p>10.4.2 Safety Considerations</p> <p>10.4.2.1 The EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling any dewar.</p>	

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10.4.2.2	During filament replacement be sure to disable the beam power supply located on the back panel of the beam supply under the bias knob.	
10.4.2.3	Be aware of elevated temperatures when changing a filament which has been in operation.	
10.4.3	Minimum Standards and Controls	
10.4.3.1	The Cu L α peak should be symmetrical through the peak centroid line as should the Cu K α peak. The centroids of these peaks will be monitored during the automated run. As a matter of QA these peaks should be within 0.03 KeV of the nominal values. FWHM of Cu K α will also be monitored during the automated run. It should not exceed 185 eV.	
10.4.3.2	Before an automated GSR run can begin the SEM filament must be saturated at 20 kV and aligned with the manual gun centering knobs in the alt S mode on an area of the sample holder where beam damage will not occur.	
10.4.3.3	A positive control consists of a sampling device containing known primer residue particles and iron spheroids and a copper strip.	
10.4.3.4	The backscatter detector threshold is set to detect GSR particles \geq 1 micron in diameter.	
10.4.3.5	A QC analysis is run on the same area on the positive control at the beginning of the run, after 3 samples have been examined and at the end of the run. After the run is complete 8 to 10 particle images and their corresponding spectra from each of the positive controls is generated showing 1.0 micron diameter GSR particles. These sheets are stored in a QC notebook in the SEM lab.	
10.4.3.6	In every automated SEM/EDS GSR run, a negative control is analyzed just before the final positive control field. The negative control is the first sample to be loaded in the SEM and the last sample to be removed after the run is complete. Negative control samples can be reused providing they have not been carbon coated. On or about the 1 st of each month the current negative control sample will be discarded. A carbon coated negative control sample will be discarded after the automated GSR run due to loss of adhesive properties during the carbon coating procedure.	
10.4.3.7	Approximately once a month the photographic unit of the SEM is checked using an ASTM measurement standard and a gold standard on carbon. Apertures are replaced if the image can't be stigmated.	
10.4.4	Analytical Procedures	
10.4.4.1	A GSR analysis setup is performed by deleting data from the last run.	
10.4.4.2	A new automated run is setup. DHDGSR1.RPF, .ZRR and .VEC files are selected. QA analysis is run in position A and typically after every 3 case samples as well as after the negative control at the end of the run. Once the setup has been created it is printed out and saved.	
10.4.4.3	Load samples into SEM as described in GSR analysis setup sheet.	
10.4.4.4	The SEM is pumped down to operational vacuum.	
10.4.4.5	The filament is saturated at 20 kV and allowed to stabilize for at least 15 minutes.	
10.4.4.6	The beam is placed on a copper strip and the spot size adjusted to achieve 6,000 CPS or a maximum of 40% Dead Time. A spectrum from 0 to 10.24 KeV is collected until VFS reaches a minimum of 8000. Peak centroids should be symmetrical as described in Minimum Standards and Controls (§ 10.4.3).	

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10.4.4.7	The backscatter electron detector is set to detect particles ≥ 1.0 micron in diameter in the following manner: The stage is driven to the QA field on the GSR standard sample. An area is located where 1.0 micron particles are visible at 1000X in the right hand image. This image is focused and stigmated using the mini imaging and medium scan speed mode. Imaging is then switched to normal and high scan speed. A particle approximately 1.0 micron in diameter is centered in the crosshairs in the right hand image. The resultant intensity should be 91 or greater. This is achieved by keeping the brightness at -0.01 and adjusting the contrast accordingly.	
10.4.4.8	A linescan is performed across the copper strip and GSR STD at 100X. The background height should be below detection level. Typically peak to trough should be visible along the bottom of the scale. The waveform across copper should be visible with the peaks just above the adjustable reference marker line when the reference threshold is set at 220 (Dwell time 4 μsec).	
10.4.4.9	Each sample to be run is then checked using a four point focus system through the stage setup program. It is imperative that the sample coordinate positions are defined in <u>exactly</u> the same order as listed on the GSR setup sheet. Areas of the stub where the aluminum surface is exposed should be avoided.	
10.4.4.10	The magnification is returned to 100X. The area containing the 1.0 micron particles on the GSR STD is focused as described in ¶ 10.4.4.7 and moved to the upper left hand corner of the left imaging field at 100X. This position is now stored in the stage setup for the QA field in position S.	
10.4.4.11	The automated run is started.	
10.4.4.12	The GSR STD. Field is monitored to ensure 1.0 micron particles are being detected. If they are not, the backscatter image and/or focus must be readjusted.	
10.4.4.13	When the automated run is finished (typically the following day) a calibration is done on copper to check the EDS detector as described earlier.	
10.4.4.14	The SEM/EDS parameters are set up as listed on the "GSR Worksheet SEM/EDS".	
10.4.4.15	The operator performs a manual confirmation of the potential GSR particles detected during the automated run.	
10.5 Automated SEM/EDS GSR Analysis, ASPEX (Formerly R. J. Lee) PSEM2000VP		
10.5.1	Purpose	
10.5.1.1	Automated SEM/EDS is used to search samples for potential primer residue particles. This instrument searches a user defined area at a magnification of 500X on each sample. Generally speaking, a primer residue particle is defined as a molten or spherical particle containing the elements: Pb, Ba and Sb. Particles indicative of primer residue would have similar morphology and include 2 of the 3 elements listed above. This instrument can be operated in either high vacuum mode for electrically conductive samples or in variable pressure mode at 0.2 Torr for nonconductive samples.	
10.5.1.2	This is a sensitive, non-destructive technique that is specific for gunshot residue.	
10.5.2	Safety Considerations	
10.5.2.1	The EDS detector system must be cooled with liquid nitrogen. Insulated gloves and safety glasses shall be worn when filling any dewar.	
10.5.2.2	During filament replacement be sure to disable the beam power supply located on the back panel of the beam supply under the bias knob.	

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10.5.2.3	Be aware of elevated temperatures when changing a filament which has been in operation.	
10.5.2.4	If operating in variable pressure mode, change to high vacuum mode before changing the filament. In addition, always turn the beam off before changing vacuum modes.	
10.5.3	Minimum Standards and Controls	
10.5.3.1	The Cu L α peak should be symmetrical through the peak centroid line as should the Cu K α peak. The centroids of these peaks will be monitored during the automated run. As a matter of QA these peaks should be within 0.03 KeV of the nominal values. FWHM of Cu K α will also be monitored during the automated run. It should not exceed 185 eV. If any of these values are exceeded recalibrate the EDS detector.	
10.5.3.2	Before an automated GSR run can begin the SEM filament must be saturated at 20 kV and aligned with the manual gun centering knobs in the Ctrl right click mode on an area of the sample holder where beam damage will not occur. Also electronically center the filament by monitoring the SEI brightness and contrast of the stage surface in the line scan mode. Adjust the filament centering in the advanced filament application.	
10.5.3.3	A positive control consists of a sampling device containing known primer residue particles.	
10.5.3.4	The backscatter detector threshold is set to detect GSR particles \geq 1 micron in diameter.	
10.5.3.5	A QC analysis is run on the same area on the positive control at the beginning of the run, after 3 samples have been examined and at the end of the run. After the run is complete 10 particle images and their corresponding spectra from each of the positive controls is generated showing 1.0 micron diameter GSR particles. These sheets are stored in a QC notebook in the SEM lab.	
10.5.3.6	In every automated SEM/EDS GSR run, a negative control is analyzed just before the final positive control field. The negative control is the first sample to be loaded in the SEM and the last sample to be removed after the run is complete. Negative control samples can be reused providing they have not been carbon coated. On or about the 1 st of each month the current negative control sample will be discarded. A carbon coated negative control sample will be discarded after the automated GSR run due to loss of adhesive properties during the carbon coating procedure.	
10.5.3.7	Approximately once a month the photographic unit of the SEM is checked using an ASTM measurement standard and a gold standard on carbon. Apertures are replaced if the image can't be stigmated.	
10.5.4	Analytical Procedures	
10.5.4.1	A GSR analysis setup is performed by setting up or using an existing .afa file.	
10.5.4.2	A new automated run is setup. DHD1.ZRR and GSRCHEM.VCF files are selected. QC analysis is run in Analysis 1 position and typically after every 3 case samples as well as after the negative control at the end of the run. Once the setup has been created it is printed out and saved.	
10.5.4.3	Load samples into SEM as described in GSR analysis setup sheet.	
10.5.4.4	The SEM is pumped down to operational vacuum.	
10.5.4.5	The filament is saturated at 20 kV and allowed to stabilize for at least 15 minutes.	

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10.5.4.6	The beam is placed on a copper std. and the spot size adjusted to achieve 3,000 CPS or a maximum of 45% Dead Time. A spectrum from 0 to 10.24 KeV is collected until VFS reaches a minimum of 8000. Peak centroids should be symmetrical as described in Minimum Standards and Controls (§ 10.5.3).	
10.5.4.7	The backscatter electron detector is set to detect particles ≥ 1.0 micron in diameter in the following manner: The stage is driven to the QA field on the GSR standard sample. An area is located where 1.0 micron particles are visible at 1000X in the right hand image. This image is focused and stigmated using the mini imaging and medium scan speed mode. Imaging is then switched to normal and high scan speed.	
10.5.4.8	Magnification is dropped to 250X and the threshold image is acquired. Particles in the 1.0 micron size range must be in the green detection area. This is achieved by keeping the brightness at -1% (average noise at 32 in linescan mode) and adjusting the contrast accordingly.	
10.5.4.9	Each sample to be run is then checked using a three point focus system through the stage setup program. It is imperative that the sample coordinate positions are defined in <u>exactly</u> the same order as listed on the GSR setup sheet. Areas of the stub where the aluminum surface is exposed should be avoided.	
10.5.4.10	The magnification is returned to 250X. The area containing the 1.0 micron particles on the GSR STD is focused as described in § 10.5.4.7. The quality check is reviewed and set to check copper before first, after last and every 1 sample.	
10.5.4.11	The automated run is started. Data is stored in the next sequential file.	
10.5.4.12	The GSR STD. Field is monitored to ensure 1.0 micron particles are being detected. If they are not, the backscatter image and/or focus must be readjusted.	
10.5.4.13	When the automated run is finished (typically the following day) a calibration is done on copper to check the EDS detector as described earlier.	
10.5.4.14	The SEM/EDS parameters are set up as listed on the "GSR Worksheet SEM/EDS".	
10.5.4.15	The operator performs a manual confirmation of the potential GSR particles detected during the automated run.	
10.5.5	References	
10.5.5.1	Andrasko, J. and Maehly, A. C., "Detection of Gunshot Residue by Use of the Scanning Electron Microscope," <u>Journal of Forensic Sciences</u> , Vol. 22, 1977, pp. 279-287.	
10.5.5.2	Basu, S., "Formation of Gunshot Residues," <u>Journal of Forensic Sciences</u> , Vol. 27, 1982, pp. 72-91.	
10.5.5.3	DeGaetano, D. H., Siegel, J. A., and Klomparens, K. L., "A Comparison of Three Techniques Developed for Sampling and Analysis of Gunshot Residue by Scanning Electron Microscopy and Energy Dispersive X-Ray Analysis," <u>Journal of Forensic Sciences</u> , Vol. 37, 1992, pp. 281-300.	
10.5.5.4	Nesbitt, R. S., Wessel, J. E., and Jones, P. F., "Detection of Gunshot Residue by Use of the Scanning Electron Microscope," <u>Journal of Forensic Sciences</u> , Vol. 21, 1976, pp. 595-610.	
10.5.5.5	Sild, E. H. and Pausak, S., "Forensic Applications of SEM/EDX," <u>Scanning Electron Microscopy</u> , Vol. 2, 1979.	
10.5.5.6	Wolten, G.M., Nesbitt, R.S., Calloway, A.R., Loper, G.L. and Jones, P.F., "Final Report on Particle Analysis for Gunshot Residue Detection," Report ATR-77(7915)-3. The Aerospace Corp., Sept. 1977.	

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<p>10.5.5.7 Gunshot Residue V2.00 from 1.12 Documentation, Operator's Manual. R. J. Lee Instruments Limited. 515 Pleasant Valley Rd., Trafford, PA 15085.</p> <p>10.6 Documentation</p> <p>10.6.1 Elements detected in potential GSR particles and the diameter of the particles are documented on the output.</p> <p>10.6.2 The original output will be retained in the case file associated with the 1st case sample in the automated run.</p> <p>10.6.3 A representative photograph and corresponding EDS spectrum is generated for the case file. If an Item has multiple sampling devices associated with it (i.e. right hand, left hand) and GSR is found on both samples, then a photo and EDS spectrum of a typical GSR particle is made from one sampling device and a hard copy of a x-ray spectrum from a typical GSR particle found on the other sampling device is also made with a photo being optional. EDS spectra should be obtained at 8K counts full scale or 99 seconds Live Time whenever possible.</p> <p>10.6.4 Spectral files and image files (where applicable) from the representative particles are saved to the hard drive and downloaded for archiving purposes.</p> <p>10.6.5 The GSR Evidence Handling Worksheet is used to provide documentation for the results reported in the Certificate of Analysis. "Boiler plate" wording on the Worksheet that is not applicable may be struck through with a single line and the entire paragraph bracketed and initialed by the examiner.</p> <p>10.6.6 The QA output must include a positive control field before and after the casefile sample/s. A copy of the negative control must also be included in the casefile or QA binder (where applicable).</p> <p>10.6.7 A stats file for each of the case samples analyzed is also included in the casefile.</p> <p>10.6.8 The GSR ANALYSIS INFORMATION FORM that is provided in the GSR kit is retained by the laboratory and kept in the casefile.</p> <p>10.6.9 The outside of the GSR kit is photocopied and retained in the casefile.</p> <p>10.6.10 In suicide cases, either the right or left hand sample is analyzed first (based upon information on the RFLE or GSR ANALYSIS INFORMATION SHEET). If more than one GSR particle is found on the sample, the second sampling device is not analyzed.</p> <p>10.7 Report Wording</p> <p>To the maximum extent possible, report wording will be selected from the following:</p> <p>10.7.1 No primer residue particles were found on the samples in Item _____ marked right hand or left hand.</p> <p>10.7.2 Particles found on the samples in Item _____ marked right/left hand were identified as primer residue.</p> <p>10.7.3 Particles found on the samples in Item _____ marked right/left hand were indicative of primer residue.</p> <p>10.7.4 The sample in Item _____ marked right/left hand was not necessary for analysis.</p> <p align="right">◆End</p>	